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UNPUBLISHED PRELIMINARY DATA

A STUDY OF BONDING BETWEEN GLASS AND PLASTIC IN GLASS-REINFORCED PLASTICS: PHASE II

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I INTRODUCTION

This report covers work done during the first quarter of Phase II of the project. Work was resumed on November 1, 1965 following a short lapse after completion of Phase I. The objective of Phase I was to investigate methods for halogenation of glass and/or silica surfaces, with formation of silicon-chlorine or silicon-fluorine bonds.

The objective of Phase II is to study methods of producing an organic "finish," on glass and quartz fibers, that will be bonded to the fibers by means of silicon-carbon bonds. This work will make use of the halogenation methods developed in Phase I.

II DISCUSSION AND EXPERIMENTAL

Halogenation Reactions

Halogenation of Particulate Materials

Particulate materials such as ground silica or glass beads, because of their high surface-to-weight ratio, will be used to develop lower-temperature halogenation procedures, and for evaluation of the effect of surface treatment upon bonding to plastics.

An improved apparatus was built for carrying out halogenations of particulate material under conditions of controlled and uniform temperature.

Chlorination of finely divided low-iron silica gave erratic values of chlorine-atom population as a function of temperature of reaction. An investigation revealed that the surface area of these particles, as measured by nitrogen adsorption, increased about tenfold when the degassing temperature was increased from 150°C to 300°C. The variable surface area appears to be due to small cracks produced by crushing.

Gases adsorbed in these cracks may not be removed at temperatures sufficient to desorb "surface" layers. If this is the case, the true, or exterior, surface of these materials cannot be accurately determined. Therefore, this phase of the work will be continued when a source of beads (fire-polished) is located.

Halogenation of Large-Diameter Cylinders

An apparatus has been designed for halogenation and subsequent alkylation of glass or silica cylinders of about 1.75-in. diameter and up to 6 in. long. These cylinders, after surface treatment, will be cut into 1.5-in. lengths and used to evaluate adhesion by the peel-test method. This apparatus should be ready for use early in March.

Evaluation Procedures

Three methods have been chosen for preliminary evaluation of the effect of surface treatment of glass (and silica) upon adhesion to polymers. They are: sonic tests, dilatometric tests, and peel tests.

Sonic Tests. The sonic test (absorption of sound) allows a comparison of the degree of bonding which occurs in a series of samples. This method will be applied to small glass beads embedded in poly (ethyl methacrylate) and other types of elastomers. The measurement and interpretation will be carried out by Mr. John Martner, of the Institute's Sonics Department.

Dilatometric Test. The dilatometric test is based upon the change in volume that may occur when a stress is applied to a compliant composite material. If the composite material is sufficiently strained (e.g. in tension), stress concentrations will result in internal rupture either within the bulk of the elastomer, due to cohesive failure, or at the interface, due to adhesive failure. Such rupture results in an increase in total volume of the sample. Measurement of the strain required and the degree of failure (related to the volume increase, as measured in

a dilatometer) may providee useful data on adhesion between reinforcement and polymer in composite materials.

Both sonic and dilatometric methods will be applied to the evaluation of bonding between particulate glass or silica fillers in polymer matrices. As discussed under Halogenation Procedures, a nonporous, crack-free material such as beads (with a fire-polished surface) is required. The effect of surface modification upon adhesion will be studied with poly (ethyl methacrylate) as an initial elastomer. The success of these methods with this system will guide work with other polymers.

Peel Test. A modified peel-test apparatus was designed to compare the effects of surface treatment on the adhesion to cylindrical glass surfaces. Glass cylinders of about 1.75 in. OD x 1.5 in. long may be accommodated. A Baldwin-Universal Tester will be used to measure the force required to peel commercial and experimental tapes bonded with adhesives representing desirable chemical types (e.g., phenolic, epoxy, etc.) of polymers.

The construction of the peel-test apparatus should be completed sometime in March.

III FUTURE WORK

Work plans for the second quarter call for: completion of apparatus for treatment and peel-testing of cylindrical glass and silica surfaces; and initiation of sonic-, dilatometric-, and peel-test programs on surface- treated glass and silica.

Respectfully submitted,

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